

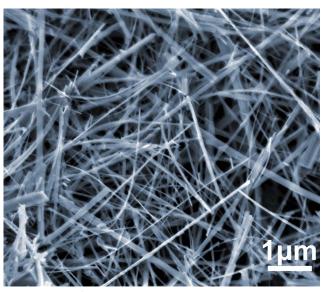
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#### **ON THE COVER**

SEM image of the pristine TiO, nanobelts.

netlog is a quarterly newsletter, which highlights recent achievements and ongoing research at NETL. Any comments or suggestions, please ula Turner at paula.turner@netl.doe.gov or call 266.

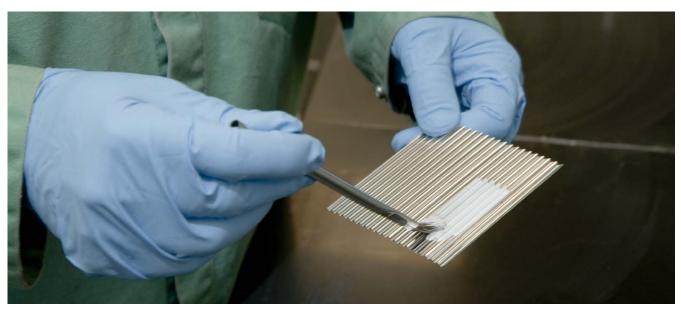


SEM image of the pristine TiO, nanobelts.

#### Nanomaterials Research Published

Photocatalytic materials are an important new class of materials with potential for use in environmental purification, hydrolysis of water, and CO<sub>2</sub> and NO<sub>2</sub> reduction. Recent experiments combined with density functional theory (DFT) calculations have been performed to better understand the underlying photocatalysis mechanisms of nitrogen-doped titania nanobelts. Nitrogen doping shifts the photocatalytic activity from the ultraviolet to the visible spectrum, which is advantageous for a number of applications. This research, which was a collaborative effort by WVU researchers Professor James P. Lewis, Professor Nick Wu and students, and NETL researcher Dr. A. Manivannan, was recently published as an article entitled "Origin of Photocatalytic Activity of Nitrogen-Doped TiO<sub>3</sub> Nanobelts" in the prestigious Journal of the American Chemical Society, 2009, 131, 12290-12297.

Contact: <u>A. Manivannan</u>, 304-285-2078 ext 620, Energy System Dynamics



The cerium oxide/activator slurry is painted onto the surface of a solid oxide fuel cell interconnect plate. The slurry has also been applied by dipping and spray coating with equally good results.

#### **Cerium Diffusion Coating Patent Awarded**

The U.S. Patent and Trademark Office recently awarded a patent for "Method of Applying a Cerium Diffusion Coating to a Metallic Alloy" to NETL researchers Dr. Paul D. Jablonski and Dr. David E. Alman. The NETL-developed technology provides a simple method for improving the oxidation resistance of chromia-forming alloys used in fossil energy applications. The technique involves slurry coating the surface of the alloy with a mixture of cerium oxide and a halide activator, followed by a thermal treatment which pre-oxides the surface. The treatment has been found to enhance the oxidation resistance of numerous commercial alloys by incorporating cerium or other rare earth elements into the surface. The simple technique can be applied to flat or curved structures, including the interior surfaces of tubes, and has been applied to numerous alloys of interest for fossil energy applications, including: ferritic steels for interconnects for SOFCs, such as type 441ss and Crofer APU; and martensitic steels, austenitic steels, and nickel-base alloys for advanced USC boiler and turbine applications, such as P91 and Save 12; Type 347, and H 230. In most cases, the surface treatment improves oxidation resistance by a factor of 2 to 3, and in a few alloys it can lead to an order of magnitude improvement in performance.

Contact: Paul Jablonski, 541-967-5982



#### NETL Captures Four R&D 100 Awards

Following years of development, four NETL technologies have captured R&D Magazine's prestigious R&D 100 awards, emblematic of the top 100 innovations for 2008. The four NETL recipients include technologies to remove gases, including mercury, from power plants, a simulation process to efficiently and cost-effectively help to design power plants, and a technology to detect underground carbon dioxide (CO<sub>2</sub>) leakage from geologic storage formations. The winning technologies are the following:



#### **SEQURE™ Tracer Technology**

This patent-pending technology uses perfluorocarbon tracers, or PFT's, to ultra-sensitively detect  $\mathrm{CO}_2$  leakage from geological storage reservoirs. Since the capture and permanent storage of  $\mathrm{CO}_2$  is vitally important to addressing greenhouse gas emissions, the Office of Fossil Energy must have technology available to verify that  $\mathrm{CO}_2$  is not leaking from deep storage reservoirs in the nation's carbon sequestration program. This technology has been tested at several pilot-scale sites and has proven to be successful in detecting  $\mathrm{CO}_2$  in field simulation experiments.

Contact: Diane Newlon, 304-285-4086



### Thief Process to Remove Mercury From Flue Gas

Researchers at NETL developed the Thief Process, which extracts partially burned coal from a pulverized coal-fired combustor using a suction pipe, or "thief," and injects the resulting carbonaceous material into the flue gas to remove the mercury. The process greatly reduces the costs of removing mercury by using already existing coal rather than expensive activated carbon. The process can prevent 90 percent of the mercury from reaching the atmosphere, thereby making the air safer for people and generating clean energy from domestic coal. The process was licensed to Nalco Mobotec, which began marketing it in December 2008.

Contact: Evan Granite, 412-386-4607

#### Clay-Liquid CO, Removal Sorbent



In cooperation with Sud-Chemie of Louisville, Kentucky, NETL developed a low-cost, solid-state sorbent that removes CO<sub>2</sub> from power plant flue gas and other gases. The sorbent's low cost, availability,

and simple preparation contribute to a significant reduction in total energy costs when compared to currently used commercial processes associated with carbon capture. The end result is that operators can continue to burn coal to provide low-cost electric power to consumers.

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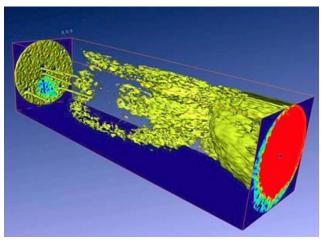


### VE-PSI: Virtual Engineering Process Simulator Interface

The VE-PSI software provides engineers with the ability to design and optimize power plants within a virtual engineering environment. Engineering data from process simulation, computational fluid dynamics, and computer-aided design can be seamlessly integrated and easily analyzed within an immersive, interactive, three-dimensional power plant walk-through system. VE-PSI enables engineers to create virtual prototypes of new plant designs more quickly, more efficiently, and at less cost than ever before, as well as improve existing designs before expending time and materials on physical prototypes and pilot plants. Developed by NETL in conjunction with Ames Laboratory, Ames, Iowa, and Reaction Engineering International of Salt Lake City, Utah, this process has been shown to save time, energy, and costs related to plant design and ultimately benefit the energy-consuming public.

Note: This technology also received the Federal Laboratory Consortium's Mid-Atlantic Region 2009 Excellence in Technology Transfer Award. The award was presented to NETL researchers Dr. Stephen Zitney and Terry Jordan at the FLC's annual Mid-Atlantic Regional Meeting on September 17, 2009, in Gettysburg, Pennsylvania.

Contact: Stephen Zitney, 304-285-1379



The middle yellow body is the surface of the hydrate concentrated area. The square with the red area on the right shows the concentration of hydrate on that side; red means higher saturation of hydrate.

### X Ray CT Images Capture Methane Hydrate Formation

Dr. Yongkoo Seol and his gas hydrate laboratory team at NETL have formed methane hydrate in a sandy porous media and have successfully captured real-time X-ray computed tomography (CT) images. The experiment was designed to study behavior of methane hydrate under various conditions potentially encountered during methane production and natural hydrate formation. The CT images clearly show patterns of hydrate distribution during the hydrate formation and dissociation within the porous media under high pressure (≈1600 psig) and low temperature  $(\approx 8 \, ^{\circ}\text{C})$ . Dr. Seol's team is developing a computer program to convert the CT images into model grids for numerical simulation studies. His team is working closely with both the CT scanner project and CO<sub>3</sub> sequestration project teams. This collaboration made the difficult work both efficient and seamless.

Contact: Yongkoo Seol, 304-285-2029 ext. 605

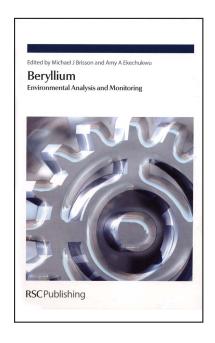


Researcher examines NETL-developed and improved compositions of chrome-oxide refractory materials for severe service environments.

### Refractory Liner Compositions Identified for Slagging Gasifiers

Refractory companies have worked with the laboratory to evaluate and improve existing commercial compositions. Rotary slag testing of 14 of these refractory compositions in flowing gasifier slag at 1600 °C for 5 hours has identified five with potential for use in slagging gasifiers. Discussions are underway with commercial gasifier users to evaluate in field trials those compositions with promise of extending gasifier on-line service life. Slagging gasifiers are lined with high chrome oxide refractory materials that must be replaced every 3-36 months, a service life that depends on gasifier environment (temperature and pressure), the amount and type of carbon feedstock, and the amount of cycling between room and gasification temperature. Refractory wear occurs by two means, corrosion and spalling. The current service life does not meet the performance needs of gasifier users, and limits achieving an online gasifier availability of 85-95% for utility applications and greater than 95% in applications such as chemical feedstock production. Failure to meet these criteria has created a potential roadblock to widespread acceptance and commercialization of advanced gasification technologies and is a reason refractory liners were identified as a key barrier to widespread commercialization of gasification technology.

Contact: James Bennett, 541-967-5983



### NETL Safety Specialist Writes Chapter in Book on Beryllium

As the use of beryllium grows worldwide, the need for a single source of information on this important but toxic element is of increasing importance. This comprehensive book describes all aspects of the current sampling and analysis techniques for tracelevel beryllium in the workplace. It offers both a historical perspective and a description of the state-of-the-art in a single book.

Published in September by the Royal Society of Chemistry, the book is entitled "Beryllium Environmental Analysis and Monitoring" and includes a chapter on surface sampling written by NETL employee Glenn Rondeau.

Contact: Glenn Rondeau, 541-967-5883



Eight sensor pig in a test pipe.

### Sensor Developed to Detect Defects in Buried Plastic Natural Gas Pipelines

Researchers have developed the first technology to detect flaws in plastic natural gas pipelines without disrupting pipeline operations. This innovative technology can inspect plastic pipelines for small-scale voids, cracks, holes, and defects.

Over 500,000 miles of polyethylene plastic natural gas pipeline (PE) has been installed over the past 45 years in North America, and a need exists to inspect these pipes for brittle cracking, excavation damage, and other safety hazards. A research team at NETL has developed an innovative flexible sensor that conforms to the pipe wall using flexible circuit board materials.

A multi-layer laminate made of alternating layers of copper sheet and Kapton (a material developed by DuPont that can remain stable in a wide variety of temperatures) is fabricated with circuit traces and sensor electrodes chemically etched into the copper sheet. Then, it is sealed together with pressure and adhesive to form a flexible sensor. A negative photo-lithographic technique is used to create the electrode shapes and circuit wiring on the individual levels, thereby ensuring uniformity from sensor to sensor. This new sensor is compact, thin, and flexible allowing it to conform to the inside of the pipeline wall surface with minimal air gap to ensure accurate capacitive sensing. NETL's capacitance sensor projects an electric field into the wall of a plastic natural gas pipeline to sense internal or external damage.

Plastic can be tested for the presence of cracks and voids by using capacitance measurements taken against the pipe wall. This capacitive sensor technology is now able to analyze the entire 360° circumference of a pipeline wall from the inside. The newest discoveries include a flexible sensor that can adapt to the shape of the inner pipeline wall and a multi-sensor pig that can traverse the inside of a pipeline without excavating. The platform (the pig) used to inspect the inside of a pipelinehas been developed to allow multiple sets of eight sensors to be deployed. The sensors are 45° apart and are projected into contact with the pipe wall using a spring-loaded suspension arm that can retract automatically if a PE weld seam or pipe discontinuity is encountered. Additional sets of eight sensors can be added by utilizing the modular pig design. This design allows the capacitance of the entire 360° pipe wall circumference to be measured and recorded. Additional sensors can also be used to provide redundant data or greater data precision.

The team of researchers has already published one paper and a new paper has been submitted for publication in the *Review of Scientific Instruments*.

A U.S. patent application, "Capacitance Probe for Detection of Anomalies in Non-Metal Plastic Pipe," was filed in December 2007. Another patent was initiated in July 2009.

An innovative flexible sensor combined with a multi-sensor inspection pig provides an inspection technology that has no known comparisons in the industry. Advancing this technology while simultaneously lowering costs will protect pipeline infrastructure investment and increase public safety.

Contact: Mahendra Mathur, 412-4605



### NETL Scientist Elected Fellow of ASM International

Dr. David Alman, Director of NETL's Materials Performance Division of the Office of Research & Development, was elected by the Board of Trustees of ASM International as a Fellow of the society. ASM International is one of the preeminent worldwide materials science and engineering technical societies. The society was formerly known as the American Society for Metals until its name change in the 1990s to ASM International. ASM established the Fellow of the Society in 1969 to provide recognition to members for their contributions to the materials science and engineering field. Dr. Alman was recognized by the Society for his contributions to the field of materials science and engineering for the development and design of novel materials and surface structures for power generation and high temperature applications.

Contact: David Alman, 541-967-5885



# AIChE Selects NETL Researcher for Prestigious Award

Dr. Madhava Syamlal, NETL Focus Area Leader, Computational and Basic Sciences, has been selected to receive the 2009 American Institute of Chemical Engineers (AIChE) Particle Technology Forum Fluidization Process Recognition Award, presented biennially for engineering leadership and significant contributions to the science and technology of fluidization or fluidized processes. The award presentation will take place November 11 at the Annual AIChE Meeting in Nashville, Tennessee. Dr. Syamlal was also the recipient of one of Illinois Institute of Technology's IIT 2009 Professional Achievement awards earlier this year.

Contact: Madhava Syamlal, 304-285-4685

# Co-gasification Test Matrix Completed

Researchers recently completed a test matrix focused on developing an understanding of the influence of biomass on coal-gasification reactions. A series of Illinois #6 and Switchgrass (100, 85, 70, 50, and 0 wt% coal-balance biomass) tests were conducted at conditions consistent with a moving-bed or fixed-bed gasifier, and results suggest that the addition of biomass influences several characteristics of the gasification reactions. Specifically, the addition of biomass increases the rate of the pyrolysis reaction, increases the conversion of coal, and reduces the amount of tars produced.

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## Paper Describes Viscosity of Fe<sub>2</sub>O<sub>3</sub> - Deionized Water Nanofluids

A paper by NETL researchers on some experimental observations on the effects of the shear rates and particle volume fractions on the shear stress and the viscosity of Fe<sub>2</sub>O<sub>3</sub> – deionized water (DW) nanofluids with polyvinylpyrrolidone (PVP) or polyethylene oxide (PEO) as a dispersant has been published in the *International Journal of Thermal Sciences.* The paper is entitled "Experimental observations of the effects of shear rates and particle concentration on the viscosity of Fe<sub>2</sub>O<sub>3</sub> - Deionized water nanofluids," Vol. 48(2009), pp. 1294-1301. The results of this study demon-strate that these fluids have yield stresses and behave as shear-thinning non-Newtonian fluids. The findings also indicate that these fluids have yield stresses that depend on the particle volume fraction. The NETL researchers have been involved with the formulation of constitutive relations and the numerical simulation of a variety of engineering problems dealing with complex fluids such as drilling fluids.

Contact: <u>Tran X. Phuoc</u>, 412-386-6024 and <u>Mehrdad Massoudi</u>, 412-386-4975

#### New Material Characterization Capability for Fuel Cell Characterization

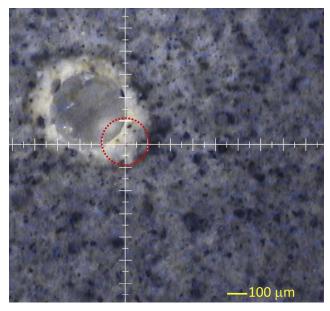
As part of NETL's University Research Initiative, a team from NETL and WVU has developed a new experimental capability and mathematical analysis method for electronic conductivity relaxation (ECR) material characterization. The ECR technique will be used to obtain data on the surface oxygen exchange rate and oxygen bulk diffusion coefficients of mixed electronic and ionic conductors, which are two of the most important properties for solid oxide fuel cell cathodes. Compared to other techniques to obtain the same results, such as the isotope-exchange depth-profile method, ECR is easier to apply and offers significant cost advantages.

Contact: Randall Gemmen, 304-285-4536

## Solid Oxide Fuel Cell Testing Begins at PSDF Facility

The NETL mobile solid oxide fuel cell (SOFC) test stand—the Multi-Cell Array (MCA)—was recently installed at the Wilsonville, Alabama, Power Systems Development Facility in order to evaluate the effects of trace contaminants from coal syngas on the SOFC anode performance. In September, the MCA was started up on hydrogen, and all 12 cells have demonstrated good performance. Testing will continue for several more weeks as NETL researchers acquire the data necessary to determine the impact of trace contaminants on the anode performance.

Contact: Kirk Gerdes, 304-285-4342



Photomicrograph of cement core showing micro-XRD area at the bore-hole/cement interface.

#### NETL Tests Look into the Fate of Small Channels in Cements

NETL researchers are conducting flow-through tests to investigate the behavior of flow channels in well-bore cements in the presence of pressurized CO<sub>2</sub>.

During  $\mathrm{CO}_2$  injection in geological sequestration,  $\mathrm{CO}_2$  will interact chemically with well-bore cement, which is typically present in deep wells. Fractures or channels in the cement may allow the  $\mathrm{CO}_2$  to flow into other formations or to the surface.

Depending on the conditions, flow paths in cement may either be enlarged through dissolution of material or be sealed with new minerals that are deposited as CO<sub>2</sub> and brine flow through the flow paths.

Tests indicate that the closing of a flow path is possible for small channels and low flow velocity of carbonated water. These results indicate that certain geometries and sizes of physical pathways in wellbore cement may not increase the risk of  ${\rm CO}_2$  release.

In an experimental setup at NETL, brine simulating deep aquifer fluid is mixed with  ${\rm CO_2}$  and forced at high pressure through channels of varying sizes in cement cores. The pressure difference across the core is recorded continuously to judge whether the channel is getting larger or shrinking. Once the test is completed, the cores are evaluated using CT scanning, X-ray crystallography, and electron microscopy.

Contact: Brian Strazisar, 412-386-5988

# Steamside Oxidation Performance Method Used for Advanced Coal-Fired Boilers

Researchers calculate that for the highest ultrasupercritical steam conditions examined (760 °C; 35 MPa), the time-to-failure for protective chromia (Cr<sub>2</sub>O<sub>2</sub>) scales is quite short for turbine blades. The failure rate is also a concern within steam pipes and the hotter portions of superheater tubes. The finding is based on chromium (Cr) concentration profiles resulting from Cr diffusion within an alloy and the evaporation rate of Cr from Cr<sub>2</sub>O<sub>3</sub> at those conditions. Alloy additions such as titanium may mitigate the deleterious effects of breakaway oxidation by reducing the Cr evaporation rate. The NETL method, previously developed for flat plate components, is now suitable for cylindrical geometries and is described in the <u>Journal of the Electrochemical Society</u> (Vol.156 (2009), No. 9, pg. C292 ff.).

Contact: Gordon Holcomb, 541-967-5874

# Oxy-fuel Turbine Technology Captures CO<sub>2</sub> More Economically

A promising technology to generate electricity from turbines powered from oxygen-fired fuels will allow CO<sub>3</sub> capture to occur more economically than using airfired fuels. The intermediate pressure turbine, which operates at the highest temperatures in the proposed system, will at this stage of development utilize existing gas turbine technology. However, the oxy-fuel turbine steam-CO<sub>3</sub>-oxygen environment is different, and perhaps more aggressive, than gas turbine environments. Materials performance of nickel-base and cobalt-base superalloys and superalloy/coating systems are being evaluated in the oxy-fuel turbine environment to qualify them for use. Evaluated properties include oxidation behavior and low-cycle fatigue resistance. Researchers have completed 1000-hour exposures in H<sub>2</sub>O-10%CO<sub>2</sub>-0.2%O<sub>2</sub> at 630, 693, 748, and 821 °C of oxidation, and low-cycle fatigue (LCF) specimens. The oxidation behavior is being examined using microstructural evaluation at NETL. Siemens is examining LCF behavior as part of the Zero Emissions Coal Syngas Oxygen Turbo Machinery cooperative agreement.

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